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The reaction flask was then maintained at 100°C for a total of 6 hours then cooled down to ambient laboratory temperature. Next the contents of the flask were transferred into 2 litres of methanol containing 10% de-ionised water in an ice-cooled 5 litre beaker. Immediately an off-white solid precipitate came out of solution, this was filtered off and collected. The crude solid was recrystallised a total of 4 times from a tetrahydrofuran/methanol solution producing a white solid product.

The product was obtained in a quantity of 31.5g which was a 37% yield. It had a melting point of 110°C.

- 15 Step 2, partial de-acylation
- Glacial acetic acid (2.04g) was added slowly dropwise with stirring into a solution of ethylenediamine (4.09g) in tetrahydrofuran (THF, 850cm³). A white precipitate formed which remained during the reaction. α -Cellobiose
- 20 octanonanoate (50g) was then added and the whole reaction mixture stirred at room temperature for a total of 48 hours.

At the end of the reaction period, the contents of the flask were transferred to a two litre separating funnel, 350cm³ of water was added and the mixture extracted with dichloromethane (250cm³). The organic layer was collected and further washed with successive 350cm³ portions of (1) dilute HCl (0.1M), (2) aqueous sodium bicarbonate (1M) and (3) water.

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The resultant organic phase was recovered, dried over anhydrous magnesium sulphate, filtered and the remaining solvent removed by rotary evaporation. A slightly sticky off-white crude solid was obtained. This was then re-crystallised from a mixture of THF/methanol (50:300cm³). During overnight storage, a white solid precipitated out and was filtered off, dried and collected, yielding 30.5g of a white free-flowing solid as intermediate product (68% Yield).

Step 3.

3A - Re-acylation with an acyl chloride

This route is exemplified for the benzoate ester, and is useable for all the esters by substituting the other acid chlorides for benzoyl chloride.

A 3 neck 500cm³ round bottomed flask was charged with cellobiose heptanonanoate (5g, 3.78×10^{-3} moles) together with 125cm³ of toluene. The mixture was stirred thoroughly until a clear solution resulted. Next triethylamine (0.479g, 4.73×10^{-3} moles) was slowly added dropwise to the solution.

Thereafter, Benzoyl Chloride (0.665g, 4.73×10^{-3} moles) was added slowly and cautiously via a pressure equalising dropping funnel into the reaction mix. When addition of the reagents was complete, the whole reaction solution was heated up to and maintained under reflux conditions for a total of 2-3hrs. The flask was then removed from the heat and after cooling was filtered to remove the solid triethylamine hydrochloride salt present. A clear straw

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- coloured liquid was obtained. All solvent was then removed by rotary evaporation to give a crude product, a straw coloured gel-like material. The crude product was re-crystallised from THF-MeOH (20cm³:120cm³). The resultant
- 5 product, a white free-flowing solid, was filtered off, collected and dried at 40-45°C. Yield was 3.5g (65%)

3B Re-acylation employing an acid/catalyst

- This method is exemplified using benzoic acid and can also
- 10 be used for making the other cellobiose esters by replacing benzoic acid by the appropriate acid.

- A 2 neck 250cm³ round bottomed flask was charged with Benzoic Acid (29.54g, 0.24moles) and trifluoroacetic
- 15 anhydride(19.05g, 0.091moles). The mixture was stirred and heated to and maintained at 100°C for one hour. Cellobiose heptanonanoate (5g, 3.78x10⁻³moles) was introduced slowly via a solids addition funnel into the activated solution. After it had added completely, the reaction mixture was maintained
- 20 at 100°C stirred for a total of 6 hours. The reaction flask was then cooled down to room temperature. An ice-cooled solution of methanol-water (400cm³ MeOH:40cm³ water) was poured into the flask, whereupon a solid precipitate formed immediately, was filtered off and re-crystallised from THF-
- 25 MeOH (20cm³:120cm³). The resultant product was filtered off collected and re-crystallised a second time from THF-MeOH to remove trace acid. The final product, a white solid, was filtered off, collected and dried at 40-45°C. The yield was 3.1g (58%)